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A New Aqueous Solvent Based on a Blend of N-MethylDiEthanolAmine and TriEthylene TetraAmine for CO₂ Recovery in Post-Combustion: Kinetics Study

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Abstract

To struggle against global warming, CO₂ emissions should be limited. The improvement in the thermal efficiency of industries and buildings will contribute to reduce the CO₂ emissions for each energy unit. But, given the emergency of the situation, it will also be necessary to recover CO₂ from more concentrated sources like power plants and store it deep underground. For CO₂ recovery in post-combustion, chemical solvents are put forward since they allow a high recovery rate even with a low partial pressure of CO₂ in the flue gas. This work focuses on the development of a new solvent based on a blend of N-methyldiethanolamine (MDEA) and triethylene tetramine (TETA), an amine with four amino groups. CO₂ absorption has been investigated between 298 and 333 K using a Lewis cell with a constant interfacial area. Several concentrations of MDEA (17.5 and 40 wt.%) and TETA (3 and 6 wt.%) have been assessed. The influence of the CO₂ partial pressure on the absorption rate was pointed out. The addition of small amounts of TETA leads to a high increase in the CO₂ absorption rates. A numerical model based on the film theory has been used to determine the rate coefficient between CO₂ and TETA for each solvent.

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Keywords: Kinetics, Absorption, Carbon dioxide, N-methyldiethanolamine, Triethylene tetramine, Numerical modelling

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1. Introduction

To struggle against global warming, CO₂ emissions should be limited. Improvement in the thermal efficiency of industries and buildings will contribute to reduce the CO₂ emissions for each energy unit. But, given the emergency of the situation, it will also be necessary to recover CO₂ from more concentrated sources like power plants and to store it deep underground. For CO₂ recovery in post-combustion, the focus has been put on chemical solvents since they allow a high recovery rate even with a low partial pressure of CO₂. Alkanolamines have already been used in industry for gas sweetening [1]. Primary and secondary alkanolamines, like monoethanolamine (MEA) and diethanolamine (DEA), react directly with CO₂ to form carbamates.

A lot of studies have been done on the kinetics between CO₂ and the most usual amines. For MEA, Versteeg et al. [2] have proposed a second order reaction rate constant based on previous works found in literacy. Aboudheir et al. [3] have used the thermolecular-kinetic model (instead of the zwitterion model) to explain the reaction between CO₂ and MEA. CO₂ absorption in aqueous solutions of MDEA has been largely studied too [2], [4–9].

The kinetics is an important characteristic when designing the absorption columns. But one must keep in mind the energy requirement for the regeneration of the solvent. A good kinetic will allow reducing the solvent flow but this benefit will be compensated by an increase in the heat duty necessary to recover the CO₂ in the gaseous phase. On the contrary, a weak kinetic, such as with MDEA, will lead to a higher solvent flow but a lower energy consumption for reversing the reaction and stripping the carbon dioxide.

This observation has led to the development of new types of amine: sterically hindered amines, like the 2-amino-2-methyl-1-propanol (AMP) [10] or amines with several amino functions [11–13]. New solvents based on blends of amines have also been proposed [14–19]. Potassium carbonate promoted by piperazine has also been studied by Cullinane and Rochelle [20–21]. The objective is to activate an aqueous solution of tertiary amine, hindered amine or potassium carbonate by a primary or secondary amine. The absorption rate of CO₂ is thus increased without degrading the characteristics of the solvent regeneration. The purpose of this study is to propose a new blend of amines based on MDEA. The activator is the Triethylene tetramine (TETA), which has four amino groups. Two concentrations for each amine have been evaluated. The mass concentration of MDEA in the aqueous solvent is either 17.5 or 40 wt.% and the mass concentration of TETA is either 3 or 6 wt.%. CO₂ absorption rates have been measured at 298, 313 and 333 K for several CO₂ loadings in the solvent. The rate coefficients between CO₂ and TETA have been determined thanks to numerical model developed by Cadours and Bouallou [8].

2. Experimental section

Material

TETA was obtained from Alfa Aesar with a technical grade purity of 60%. This product is composed of four amines: one linear, two cyclical and one with branching. The linear form accounts only for 60% of the product. Its vapour pressure is very low, lower than 0.1 Pa at 303 K. This characteristic will allow limiting the solvent losses in a CO₂ recovery process. CO₂ is provided by Air Liquide with a certified purity of 99.995 vol.%.

Experimental apparatus

The experimental apparatus (Figure 1) has been designed to measure absorption rates for a blend of gaseous components. The reactor is a Lewis cell which can withstand a maximum pressure of 1.0 MPa and a maximum temperature of 400 K. This cell is composed of a quartz cylinder and two stainless steel flanges. The cell has an inner diameter of 6.0×10^{-2} m, a constant interfacial area of $11.78 \pm 0.05 \times 10^{-4}$ m² and a volume of $303.4 \pm 0.2 \times 10^{-6}$ m³. The gas phase agitation consists on a four blades propeller. A Rushton turbine ensures the liquid phase agitation. It is composed of six blades with a 4.20×10^{-2} m diameter. Not to interfere with the reactor volume, the agitators are pulled by a magnet located inside the cell and driven by a magnetic stirrer located outside the cell. Four polytetrafluoroethylene baffles are located inside the cell to avoid vortex formation. A pressure transducer (Druck, type PTX611, range: 0–1.6 MPa) measures the evolution of the pressure in the gas phase. It is calibrated against a numerical standard from Desgranges&Huot (24610 model) and a dead weight pressure balance from Desgranges & Huot (5202S model). The uncertainties are ± 0.0001 MPa. The cell is immersed in a thermo-regulated bath. The temperature is measured inside the cell in the liquid phase by a 100 Ω platinum temperature probe. This probe is calibrated against a 25 Ω reference probe (TINSLEY Precision Instrument) certified by the Laboratoire National d'Essais (Paris). The temperature is given with a precision of ± 0.03 K.

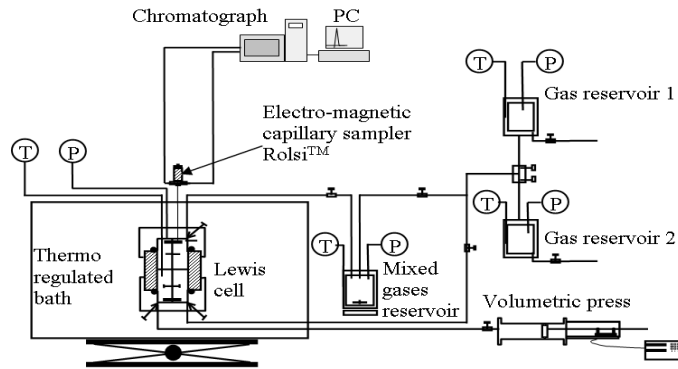


Figure 1. Experimental apparatus

Procedure

The CO₂ absorption rates can be determined thanks to the evolution of the partial pressure of CO₂ against time. The reproducibility of the absorption rates is within ±10%. The gas side mass transfer resistance is neglected. This assumption will be confirmed later.

$$\phi_{CO_2} \cdot A_{in} = -\frac{d(n_{CO_2})}{dt} = -\frac{V_G}{RT} \frac{d(P_{CO_2})}{dt} \quad (1)$$

The enhancement factor is determined by:

$$\phi_{CO_2} = \frac{E \cdot k_L}{H_{CO_2}} (P_{CO_2, in} - P_{CO_2, b}) \quad (2)$$

$$\text{With } P_{CO_2, b} = H_{CO_2} C_{CO_2, b} \quad (3)$$

The CO₂ partial pressure at the interface is calculated from the absolute pressure in the cell and the vapour pressure of the unloaded solvent:

$$P_{CO_2} = P - P_{solv} \quad (4)$$

3. Theory

Liquid side mass transfer

The liquid side mass transfer coefficient was determined by physical absorption of N₂O in aqueous solutions of MDEA [22] between 296 and 362 K for MDEA concentrations ranging from 0 to 50 wt.%. The gas side mass transfer resistance is neglected. This assumption is reasonable because the gas phase contains mainly N₂O and the stirrer speed of the gas phase has little influence on the physical absorption rate.

A dimensionless correlation is used to represent the data (eq 5). It implies three dimensionless numbers: the Reynolds number $Re = \frac{\rho N_L D_{rush}^2}{\mu}$, the Schmidt number $Sc = \frac{\mu}{\rho D_G}$ and the Sherwood number $Sh = \frac{k_L D_{cell}}{D_G}$.

This correlation is valid for a Reynolds number ranging from 215 to 5666, a Schmidt number varying from 46 until 21710 and a Sherwood number included in the range 378 - 985.

$$Sh = 0.544 Re^{0.643} Sc^{0.346} \quad (5)$$

Rate coefficient between CO₂ and TETA

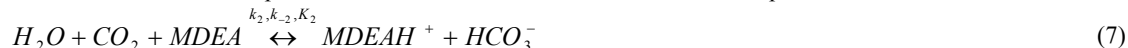
To determine the rate coefficient between CO₂ and TETA, it is necessary to assume a chemical reaction between these two compounds. The difficulty comes from the number of amino groups of the TETA. The reaction of CO₂ with a primary or secondary is generally explained by the zwitterion mechanism [23-24]. This mechanism can be split in two steps: the zwitterion formation and the conversion to carbamate. With TETA, the mechanism is more complex since this amine has four amino groups. By analogy with MEA, we have considered that one molecule of

CO₂ requires two amino groups. This assumption leads to the formation of a dicarbamate. We have only considered a global reaction to explain the CO₂ reaction with TETA.



Where $R_1 = \text{C}_2\text{H}_4$

This reaction takes only into account the dicarbamate formation. The monocarbamate formation and the amine dissociation with water were not considered. For the H₂O-MDEA-CO₂ system, several reactions occur simultaneously. But given the uncertainties on the chemical reactions between CO₂ and TETA, we have only considered the reaction 7. A partial order of 1 was selected for the different components.



The numerical model developed by Cadours and Bouallou [8] has been used to determine the rate coefficient between CO₂ and TETA. It is based on the coupling between diffusion phenomena and chemical reactions applied to the modified film theory in accordance with the work of Chang and Rochelle [25] to increase its precision. The chemical reaction 7 is assumed to be reversible. The direct rate coefficient is taken from Cadours and Bouallou [8]. The reverse rate coefficient is obtained by the equilibrium constant of the reaction [26-27].

The physico-chemical properties used come from [7], [22] and [28]. The density and viscosity of the solvents were measured between 298.15 and 333.15 K (Table 1). The viscosity of aqueous TETA solutions was measured between 293 and 333 K for low amine concentrations (Table 2).

Table 1. Density and Viscosity of aqueous MDEA-TETA solutions

	ρ Density (kg m ⁻³)			μ Viscosity (10 ⁻³ Pa s)		
	298.15 K	313.15 K	333.15 K	298.15 K	313.15 K	333.15 K
MDEA 20 wt.% TETA 3 wt.%	1017.3	1010.8	999.9	2.32	1.54	1.00
MDEA 20 wt.% TETA 6 wt.%	1019.3	1012.4	1001.3	2.74	1.78	1.12
MDEA 40 wt.% TETA 3 wt.%	1036.4	1027.9	1014.4	6.55	3.79	2.11
MDEA 40 wt.% TETA 6 wt.%	1038.5	1029.3	1015.8	8.32	4.59	2.46

Table 2. Viscosity of aqueous TETA solutions

	μ Viscosity (10 ⁻³ Pa s)				
wt.% TETA	293.15 K	303.15 K	313.15 K	323.15 K	333.15 K
0	1.000	0.804	0.657	0.549	0.470
5	1.116	0.912	0.723	0.592	0.496
10	1.438	1.141	0.870	0.699	0.567

4. Results for the absorption rate

CO₂ absorption experiments were carried out between 298 and 333 K for four different compositions. The enhancement factor is calculated from eq 2. Its evolution with the CO₂ partial pressure at 313 K is displayed on Figure 2. The impact of TETA on the absorption rate is obvious for the lowest partial pressure. Whereas the enhancement factor does not vary much with the partial pressure of CO₂ for an aqueous MDEA solution, the addition of small amounts of TETA results in a significant enhancement of the absorption rates, especially at low CO₂ partial pressure.

Figure 3 compares the absorption rates of the different solvents as a function of the CO₂ loading. For comparison, the absorption rates with MDEA and MEA aqueous solutions have been added. Addition of TETA enhances significantly the absorption rate, whatever the MDEA concentration and the CO₂ loading are. The higher the TETA concentration the greater the absorption rate. At 313.4 K, for a CO₂ partial pressure of 10000 Pa and at low CO₂ loading, the absorption rate is multiplied by 2 with an aqueous solution containing 39.98 wt.% MDEA + 3.00 wt.% TETA in comparison with an aqueous MDEA solution. It is multiplied by 8 with an aqueous solution containing 17.62 wt.% MDEA + 5.95 wt.% TETA. Whatever the MDEA concentration is, the absorption rate is increased by more than 60% when the TETA concentration increases from 3 to 6 wt.%.

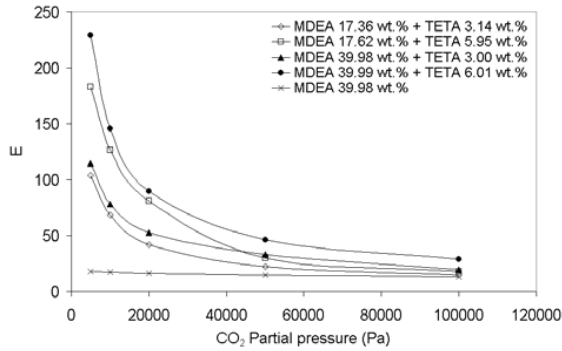


Figure 2. Enhancement factor versus CO₂ partial pressure at 313 K (CO₂ loading < 0.03 mol CO₂ / mol amines)

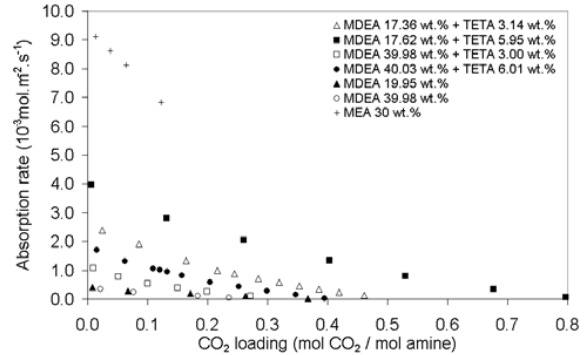


Figure 3. Absorption rate as a function of temperature and CO₂ loading (T = 313.4 K - P(CO₂) = 10000 Pa)

A high increase in the MDEA concentration, from 17.5 to 40 wt.%, has practically no effect on the absorption rate in absence of TETA, but has a negative impact when some TETA is added. Indeed, a high increase in the MDEA concentration implies a higher viscosity that cancels the benefit of a higher MDEA concentration. The amines diffuse more slowly towards the gas-liquid interface. The influence of the TETA on the absorption rate is thus weakened. At low CO₂ loading, the absorption rate is divided by 2.5 when the MDEA concentration increases from 17.5 wt.% to 40 wt.%. In comparison with an aqueous solution containing 30 wt.% MEA, the absorption rate of CO₂ with the solvents using a blend of MDEA and TETA is lower. However, it is necessary to keep in mind that, in practice, the range of CO₂ loadings will not be the same for the two types of solvent. Thus, a solvent using MEA will be used with a CO₂ loading higher than 0.15 mol CO₂ / mol MEA, limiting the absorption rate. On the contrary, the solvent using the blend of amines could work at low CO₂ loadings. While the maximum achievable CO₂ loading is about 0.25 mol CO₂ / mol MDEA for an aqueous solution of MDEA, the presence of TETA allows increasing this value, between 0.3 and 0.8 mol CO₂ / mol amines depending on the concentration of the two amines.

5. Rate coefficient between CO₂ and TETA

In absence of MDEA, some CO₂ absorption experiments were carried out for aqueous solutions of TETA between 313 and 353 K (Table 3). The average CO₂ loading is 0.228 mol CO₂ / mol TETA. The influence of this loading on the physico-chemical properties has not been taken into account.

Table 3. Raw experimental data for the CO₂ absorption in aqueous TETA solutions

wt.% TETA	T (K)	CO ₂ loading (mol CO ₂ / mol TETA)	k _L (10 ⁻⁵ m s ⁻¹)	P(CO ₂) (Pa)	Absorption rate (10 ⁻³ mol m ⁻² s ⁻¹)
4.99	313.0	0.215	2.03	10107	3.40
4.94	333.2	0.197	3.64	10194	6.58
9.99	313.2	0.267	2.12	9966	4.40
9.99	353.4	0.233	5.06	9652	1.03

The second-order rate coefficient has been expressed as an Arrhenius law (eq 8) and compared with the relation proposed by Versteeg et al. [2] for MEA and the one determined by Cadours and Bouallou [8].

$$k_1 = k_{\text{CO}_2\text{-TETA}} = 1.98 \times 10^9 \exp\left(-\frac{6315.3}{T}\right) \quad (\text{m}^3 \text{mol}^{-1} \text{s}^{-1}) \quad (8)$$

Figure 4 represents the rate coefficients against temperature. It appears that the kinetic between TETA and CO₂ is very high in comparison with MDEA. However, TETA is less reactive with CO₂ than MEA. But the chemical reaction between CO₂ and TETA considered here assumes that two molecules of CO₂ react with one molecule of TETA, whereas only half a molecule of CO₂ reacts with one molecule of MEA. The activation energy has been determined at 52.5 kJ.mol⁻¹, which is of the same order than with MEA (44.9 kJ mol⁻¹) and with MDEA (44.3 kJ mol⁻¹). The

Arrhenius law determined for TETA is a first approximation since it does not correspond to the CO₂ absorption in a fresh solution. Moreover, many physico-chemical properties are missing, such as the Henry's law constant of CO₂.

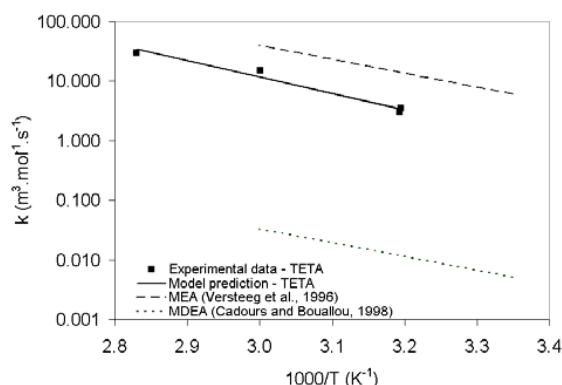


Figure 4. Rate coefficients between CO₂ and TETA, CO₂ and MEA and between CO₂ and MDEA as a function of temperature.

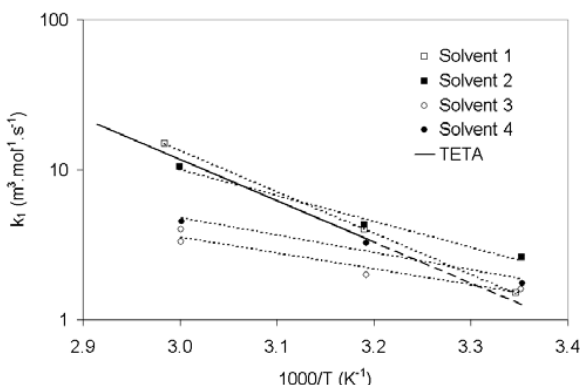


Figure 5. Rate coefficients between CO₂ and TETA in presence of MDEA as a function of temperature.

For the experiments with aqueous MDEA+TETA solutions, the rate coefficients were only determined at low CO₂ loadings (Table 4). The rate coefficient determined for each experiment is shown on Figure 5. The Arrhenius law found for the solvent 1 is very similar to the one found without MDEA (eq 8). The activation energy has been established at 52.9 kJ mol⁻¹ (eq 9).

$$k_1(\text{solvent 1}) = 2.587 \times 10^9 \exp\left(-\frac{6356.9}{T}\right) \quad (\text{m}^3 \text{mol}^{-1} \text{s}^{-1}) \quad (9)$$

For the solvent 2, the activation energy is lower than the one determined for solvent 1. It is about of 33.1 kJ mol⁻¹. The difference may come from the fact that the rate coefficient k_1 has to represent the global kinetics of the system including TETA. The chemical path used in this study may be too simple to represent correctly the behaviour between CO₂ and TETA. In this study, the complexity of the system has to be explained by only one parameter, the rate coefficient k_1 . The corresponding Arrhenius law is represented by eq 10.

$$k_1(\text{solvent 2}) = 1.535 \times 10^6 \exp\left(-\frac{3978.9}{T}\right) \quad (\text{m}^3 \text{mol}^{-1} \text{s}^{-1}) \quad (10)$$

Table 4. Raw experimental data for the CO₂ absorption in aqueous MDEA+TETA solutions

	wt.% MDEA + wt.% TETA	T (K)	CO ₂ loading (mol CO ₂ / mol amines)	k _L (10 ⁻⁵ m s ⁻¹)	P(CO ₂) (Pa)	Absorption rate (10 ⁻³ mol m ⁻² s ⁻¹)
Solvent 1	17.36 + 3.14	298.8	0.036	1.04	10025	1.62
	17.36 + 3.14	313.5	0.024	1.51	9961	2.40
	17.64 + 3.32	335.1	0.009	2.35	10089	4.22
Solvent 2	17.67 + 5.97	298.3	0.014	0.91	10036	2.78
	17.62 + 5.95	313.2	0.022	1.34	10026	3.33
	17.99 + 6.02	333.4	0.013	2.12	10064	4.70
Solvent 3	39.98 + 3.00	298.4	0.008	0.38	10001	0.87
	39.98 + 3.00	313.3	0.010	0.64	9966	1.07
	39.99 + 3.01	333.2	0.014	1.11	9934	1.45
	39.96 + 3.01	333.2	0.015	1.11	10010	1.59
Solvent 4	40.03 + 6.01	298.3	0.015	0.31	9992	1.09
	40.03 + 6.01	313.3	0.015	0.54	9986	1.71
	39.99 + 6.01	333.1	0.013	0.98	9979	2.19

The solvents 1 and 2 are those for which the rate coefficients are closer to the one found without MDEA. The weakest MDEA concentration may explain this point. The rate coefficients obtained for the solvents 3 and 4 are shifted compared to those obtained for the solvents 1 and 2. This is not due to an experimental error on the absorption rate. Indeed, for the solvent 3, the rate coefficient at 333 K has been determined with two independent experiments and lead approximately to the same result. This shift can be explained by the simplicity of the chemical reactions or by the uncertainties on the physico-chemical properties. The Arrhenius laws obtained for the solvents 3 and 4 are respectively given by the eqs 11 and 12. The activation energies are respectively 20.3 and 22.1 kJ mol⁻¹.

$$k_1(\text{solvent } 3) = 5.311 \times 10^3 \exp\left(-\frac{2436.5}{T}\right) \quad (m^3 \text{ mol}^{-1} s^{-1}) \quad (11)$$

$$k_1(\text{solvent } 4) = 1.413 \times 10^4 \exp\left(-\frac{2663.3}{T}\right) \quad (m^3 \text{ mol}^{-1} s^{-1}) \quad (12)$$

6. Conclusion

Since TETA is very reactive with CO₂, it was proposed to use this amine as an activator in an aqueous MDEA solution. The addition of small amount of TETA leads to a significant enhancement of the absorption rates in comparison with an aqueous MDEA solution. Moreover the absorption capacity of the solvent is increased. This type of solvent is a good compromise between absorption rate and efficiency of solvent regeneration. However, the behaviour of this type of solvent with oxygen must be studied, as well as the thermal degradation.

A numerical model was used to determine the rate coefficients at very low CO₂ loadings. An Arrhenius law was determined for each concentration of MDEA and TETA. We were unable to represent the whole results by only one Arrhenius law. The simplicity of the chemical system used in this study could explain this point. In addition, it was pointed out that the physico-chemical properties, such as the Henry's law constant, the solvent viscosity and the CO₂ diffusivity, have a huge influence on the determination of the rate coefficients.

Acknowledgement

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Nomenclature

A_{in} = interfacial area (m²)
 C = concentration (mol m⁻³)
 D = diffusivity (m² s⁻¹)
 D_{cell} = inner diameter of the Lewis cell (m)
 D_{rush} = diameter of the Rushton turbine (m)
 E = enhancement factor
 H = Henry's law constant (Pa m⁻³ mol⁻¹)
 k_i = rate coefficient (m³ mol⁻¹ s⁻¹)
 k_L = liquid side mass transfer coefficient (m s⁻¹)
 k_G = gas side mass transfer coefficient (mol m⁻² s⁻¹ Pa⁻¹)
 K_i = Equilibrium constant
 n = number of mole (mole)
 N = stirrer speed (rpm)
 P = pressure (Pa)
 t = time (s)
 T = temperature (K)
 V = volume (m³)
 φ = chemical absorption rate (mol m⁻² s⁻¹)

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